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(54) Title: **OXIDATION OF METAL SULFIDES USING THERMOTOLERANT BACTERIA**

(57) Abstract

A process for recovering precious or base metals from particulate refractory sulfide materials comprises: a) contacting the sulfide material with an aqueous solution containing a thermotolerant bacteria culture capable of promoting oxidation of the sulfide material at a temperature in the range from 25 to 55 °C, b) separating the oxidized residue from the aqueous liquid, and, c) treating the oxidized residue and/or the aqueous liquid to recover metal. In this context, a thermotolerant bacterium is one which has an optimum growth temperature of 40 to 45 °C, and an operating temperature of 25 to 55 °C.

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TITLE

OXIDATION OF METAL SULFIDES USING THERMOTOLERANT BACTERIA

DESCRIPTION

The present invention relates to a process for the treatment of metal containing materials by bacterial oxidation.

FIELD OF THE INVENTION

It is known that recovery of metals especially precious metals and base metals from refractory sulphide materials can be enhanced by bacterial oxidation or leaching. The bacterial treatment subjects the sulphide material to a pre-oxidation. The refractory sulphide materials can take a wide variety of forms including mineral sulphides, carbonaceous sulphide ores, sulphide flotation concentrates, sulphide gravity concentrates, sulphide tailings, sulphide mattes and sulphidic fume.

The precious metals and some base metals remain in the oxidised solid residue and can be recovered by conventional carbon in pulp or other chemical leaching processes. Some base metals such as copper, zinc and nickel go into solution and may be recovered directly by conventional solvent extraction and electrowinning.

In the past, bacterial oxidation of precious or base metal containing sulphide materials has typically been conducted using bacteria of the Thiobacillus species. However, the Thiobacillus species can only operate at temperatures up to about 40°C. Further, the oxidation effected by Thiobacillus bacteria is an exothermic reaction and it is sometimes necessary to cool the process reactors to prevent

the temperature exceeding that at which the Thiobacillus bacteria can operate.

SUMMARY OF THE INVENTION

The present invention provides a process for the bacterial oxidation of metal containing sulphide materials using thermotolerant bacteria which can operate at higher temperatures than conventional Thiobacillus bacteria.

In accordance with one aspect of the present invention there is provided a process for recovering metals from particulate refractory precious or base metal containing sulphide materials which comprises contacting the sulphide material with an aqueous solution containing a thermotolerant bacteria culture (as herein defined) capable of promoting oxidation of the sulphide material at a temperature in the range from 25 to 55°C, separating the oxidised residue from the aqueous liquid and treating the oxidised residue and/or the aqueous liquid to recover metal therefrom.

DESCRIPTION OF THE INVENTION

The thermotolerant bacteria used in the present invention are as described in "Thermophiles General, Molecular, and Applied Microbiology" edited by Thomas D. Brock and published by John Wiley & Sons (1986). In Chapter 1 of this publication, there is illustrated in Figure 1(b) a graph showing that thermotolerant bacteria grow at temperatures lower than those preferred by moderate and obligate or extreme thermophiles.

In the context of the present invention, a thermotolerant bacteria is one which has an optimum growth temperature of

40 to 45°C and an operating temperature of 25 to 55°C. Preferably, the aqueous solution used in the process of the present invention is acidic. It has been found that the optimum acidity of the aqueous liquid for growth of the thermotolerant bacteria culture used in the present invention is in the range from [redacted], whilst the optimum acidity of the aqueous liquid for operation of the process of the present invention is in the range from [redacted].

The bacterial oxidation step of the process of the present invention is conducted in the presence of nutrients which are typically dissolved salts of nitrogen, potassium and phosphorus. The nutrients may already be present in the aqueous liquid or they may be added thereto. The nutrient materials promote the growth of the thermotolerant bacteria.

It is preferred that the thermotolerant bacteria be acidophilic in view of the pH conditions under which the process of the present invention is preferably conducted. Further, the thermotolerant bacteria used in the process of the present invention are typically aerobic and thus the aqueous liquid is preferably aerated during the operation of the process to ensure that there is an adequate supply of oxygen for the bacteria. Still further, it is found that the thermotolerant bacteria culture used in the process of the present invention is typically capable of autotrophic growth. Yet further, the thermotolerant bacteria culture typically does not require additional CO_2 over and above that normally available from ambient

air.

The thermotolerant bacteria culture used in the process of the present invention may be capable of oxidising arsenic (III) to arsenic (V) in acidic aqueous solutions containing soluble iron salts. Further, the thermotolerant bacteria culture used in the process of the present invention may be capable of oxidising iron (II) to iron (III) in acidic aqueous solutions and may be capable of oxidising reduced sulphur species to sulphate ion in acidic aqueous solutions.

Also, the thermotolerant bacteria culture used in the process of the present invention is preferably capable of oxidising iron and sulphides in an aqueous liquid containing up to 20 grams/litre of sodium chloride without the addition of special nutrients or employment of the special conditions. Thus, in this case extracting the pH, temperature, oxygen, nitrogen phosphate and potassium levels are maintained as discussed above, oxidation will proceed.

Typically, a particular culture of thermotolerant bacteria contains one or more bacteria species.

The process of the present invention can be operated in heaps, dumps, agitated systems or dams.

After completion of the oxidation step the oxidised solid residue and the aqueous liquid are typically separated. In the case of precious metal recovery, the oxidised solid residue would preferably be washed and then the pH of the oxidised solid residue adjusted to a level compatible with the use of a cyanide leaching agent. Alternatively,

another reagent such as thiourea could be used under acidic conditions and so the need to adjust the pH is obviated.

EXAMPLES

The present invention will now be illustrated by the following examples.

EXAMPLE 1

A pyrite - gold concentrate designated P 1 was treated in accordance with the present invention. The concentrate contained pyrite as the major sulphide mineral with minor amounts of chalcopyrite, sphalerite, galena and arsenopyrite. Other minerals present were quartz, sericite and siderite.

The concentrate had the following assay.

Table 1

Assay of Pyrite Concentrate P 1

Element	Symbol	Assay (by weight)
Gold	Au	52.0 ppm
Iron	Fe	26.0%
Sulphur	S	27.5%
Nickel	Ni	113 ppm
Copper	Cu	880 ppm
Zinc	Zn	320 ppm
Lead	Pb	160 ppm
Arsenic	As	3750 ppm
Silver	Ag	8 ppm

Samples of the concentrate were mixed with a sulphuric acid solution at a pulp density of 3% w/w to provide a pH range of 1.2 to 1.5. Nutrients included in the acid solution

were ammonium sulphate at 200 mg/L, di-potassium hydrogen phosphate at 200 mg/L and magnesium sulphate heptahydrate at 400 mg/L.

The acid level (pH) may vary from the start value and may either rise and then fall or fall from the outset. In most tests, the variation can be significant with the final pH often less than 1.0.

The slurry was inoculated with a thermotolerant bacteria culture designated MTC 1. The inoculated slurry was shaken in conical flasks at a temperature of 43°C. Samples were removed periodically and analysed for iron and arsenic extraction to determine the progress of the treatment.

The sample was treated by bacterial oxidation for 30 days to achieve 80% oxidation of the pyrite mineral. The solids weight loss due to the oxidation process was 52%. The solid residue was then separated from the residual acid solution. Leaching of the solid residue using alkaline cyanide solution recovered 92% of the gold. In comparison, cyanide leaching could recover only 74% of the gold from the concentrate in the untreated state. These results are summarised in Table 2.

Table 2

Gold Recovery from Untreated and Oxidised Concentrate

Sample	Iron Extracted (by weight)	Gold Recovered By Cyanide Leaching (by weight)
Untreated	0%	74%
Bacterial	80%	92%

Oxidation

The cyanide solution employed to recover the gold contained sodium cyanide at a concentration of 2 g/L.

The iron in the solution from the bacterial oxidation process can be removed by adjusting the pH to above 5.0 by the addition of lime, limestone, alkaline tailings or sodium hydroxide.

The results of this test show that gold encapsulated with pyrite (FeS) can be released from the sulphide lattice by at least partial oxidation of the sulphur and iron by the thermotolerant bacteria culture MTC 1 to render the gold accessible to cyanide solution.

EXAMPLE 2

A nickel sulphide ore designated N 1 was treated in accordance with the present invention. The ore contained both sulphidic nickel and non-sulphidic nickel minerals including violarite, lizardite and niccolite (NiAs). Approximately 70% of the nickel was present as sulphidic nickel. Other minerals were siderite, goethite, pyrite, chlorite and quartz.

The ore had the following assay.

Table 3

Assay of Nickel Ore N 1

Element	Symbol	Assay (by weight)
Nickel	Ni	2.74%
Iron	Fe	18.7%

Samples of the ore were mixed with a sulphuric acid solution at a pulp density of 13% w/w to provide a pH range of 1.2 to 1.5. Nutrients included in the acid solution

were ammonium sulphate at 200 mg/L, di-potassium hydrogen phosphate at 200 mg/L and magnesium sulphate heptahydrate at 400 mg/L.

The acid level (pH) may vary from the start value and may either rise and then fall or fall from the outset. In most tests, the variation can be significant with the final pH often less than 1.0.

The slurry was inoculated with the thermotolerant bacteria culture designated MTC 1. The inoculated slurry was shaken in conical flasks at a temperature of 47°C. Samples were removed periodically and analysed for iron and nickel extraction to determine the progress of the treatment.

At the completion of the bacterial oxidation treatment, 17 days, the solution was removed from the residual solids and the residual solids washed with sulphuric acid solution to remove any residual nickel. The nickel recovery was 93% after the residual nickel was washed out of the solids residue.

The nickel could be recovered from the solution by raising the pH to a value of about 8.5, by the addition of lime or sodium hydroxide.

For comparison, the ore was also treated with iron (III) sulphate solution at pH 1.0 and 50°C for 24 hours to extract nickel. Only 16% of the nickel was recovered in this process. These results are summarized in Table 4.

Table 4

Nickel Recovery from Ore N 1

Treatment Method	Nickel in Residue	Nickel Extraction

	(by weight)	(by weight)
Iron (III) Leaching	2.03%	16%
Bacterial Oxidation	0.60%	78%
Bacterial Oxidation & Washing	0.19%	93%

The results of this test showed that base metals in ore as sulphide minerals can be recovered by the action of the thermotolerant bacteria culture MTC 1. The sulphidic minerals were oxidised to release the nickel into the acidic solution for conventional recovery.

EXAMPLE 3

A gold bearing arsenopyrite - pyrite concentrate was treated according to the present invention. This concentrate was designated AP 1. The major sulphide minerals were pyrite, 30% by weight and arsenopyrite, 35% by weight. Other minerals present were calcite, quartz and chlorite. The gold was present almost completely in the arsenopyrite.

The concentrate had the following assay.

Table 5

Assay of Arsenopyrite Concentrate AP 1

Element	Symbol	Assay (by weight)
Gold	Au	80 ppm
Arsenic	As	16.7%
Iron	Fe	28.1%
Sulphur	S	30.0%

Nickel	Ni	1.5%

Samples of the concentrate were mixed with a sulphuric acid solution at a pulp density of 3% w/w to provide a pH range of 1.0 to 1.3. Nutrients included in the acid solution were ammonium sulphate at 200 mg/L, di-potassium hydrogen phosphate at 400 mg/L and magnesium sulphate heptahydrate at 400 mg/L.

The acid level (pH) may vary from the start value and may either rise and then fall or fall from the outset. In most tests, the variation can be significant with the final pH often less than 1.0.

The slurry was inoculated with the thermotolerant bacteria culture designated MTC 1. The inoculate slurry was shaken in conical flasks at a temperature of 40°C. Samples were removed periodically and analysed for iron and arsenic extraction to determine the progress of the treatment.

The sample was treated by bacterial oxidation for 12 days to achieve 90% break down of the arsenopyrite mineral. The solids weight loss due to the oxidation process was 30%. The residual solids were then separated from the acid solution. Leaching of the separated solid residue using alkaline cyanide solution recovered 95% of the gold. In comparison, cyanide leaching could recover only 21% of the gold from the concentrate in the untreated state. These results are summarised in Table 6.

Table 6

Gold Recovery from Untreated and Oxidised Concentrate

Sample	Arsenic Extracted	Gold Recovered by Cyanide

	(by weight)	Leaching (by weight)
Untreated	0%	21
Bacterial Oxidation	90%	95

The cyanide solution employed to recover the gold contained sodium cyanide at a concentration of 2 g/L.

The arsenic and iron in the solution from the bacterial oxidation process can be removed by adjusting the pH to above 5.0 by the addition of lime, limestone, alkaline tailings or sodium hydroxide.

The results of this test show that gold encapsulated with arsenopyrite (FeAsS) can be released from the sulphide lattice by at least partial oxidation of the arsenic, sulphur and iron by the thermotolerant bacteria culture MTC 1 to render the gold accessible to cyanide solution.

EXAMPLE 4

A gold bearing arsenopyrite - pyrite concentrate was treated according to the present invention. This concentrate was designated AP 2. The major sulphide minerals were pyrite, 90% by weight and arsenopyrite 9% by weight. Other minerals present were calcite, quartz and chlorite. The gold was distributed in both the arsenopyrite and the pyrite.

The concentrate had the following assay.

Table 7

Assay of Arsenopyrite - Pyrite Concentrate AP 2

Element	Symbol	Assay (by weight)
Gold	Au	54 ppm
Arsenic	As	4.2%

Iron	Fe	35.7%
Sulphur	S	40.0%

Samples of the concentrate were mixed with a sulphuric acid solution at a pulp density of 10% w/w to provide a pH range of 1.0 to 1.3. Nutrients included in the acid solution were ammonium sulphate at 200 mg/L, di-potassium hydrogen phosphate at 400 mg/L and magnesium sulphate heptahydrate at 400 mg/L.

The acid level (pH) may vary from the start value and may either rise and then fall or fall from the outset. In most tests, the variation can be significant with the final pH often less than 1.0.

The slurry was inoculated with the thermotolerant bacteria culture designated MTC 1. The inoculated slurry was shaken in conical flasks at a temperature of 53°C. Samples were removed periodically and analysed for iron and arsenic extraction to determine the progress of the treatment.

The sample was treated by bacterial oxidation for 12 days to achieve 90% oxidation of the arsenopyrite mineral and an additional 21 days for 70% pyrite oxidation as well as arsenopyrite oxidation. The weight loss due to the oxidation process was 25% for the arsenopyrite and 78% for the 100% arsenopyrite plus 70% pyrite. The solids residue was then separated from the acid solution.

Leaching of the solid residue using alkaline cyanide solution recovered 79% of gold for the oxidation of 90% arsenopyrite and 87% for complete oxidation of the arsenopyrite and 70% of the pyrite. In comparison, cyanide leaching could recover only 53% of the gold from the

concentrate in the untreated state. These results are summarised in Table 8.

Table 8

Gold Recovery from Untreated and Oxidised Concentrate

Sample	Arsenic Extracted (by weight)	Iron Extracted (by weight)	% Gold Recovered (by weight)
Untreated	0%	0%	53%
Bacterial Oxidation	90%	25%	79%
Bacterial Oxidation	100%	70%	87%

The cyanide solution employed to recover the gold contained sodium cyanide at a concentration of 2 g/L.

The arsenic and iron in the solution from the bacterial oxidation process can be removed by adjusting the pH to above 5.0 by the addition of lime, limestone, alkaline tailings or sodium hydroxide.

The results of this test show that gold encapsulated with arsenopyrite (FeAsS) and in pyrite (FeS2) can be released from the sulphide lattice by at least partial oxidation of the arsenic, sulphur and iron by the thermotolerant bacteria culture MTC 1 to render the gold accessible to cyanide solution. This example also shows that the MTC 1 culture is able to operate according to the invention at 53°C.

The thermotolerant bacteria culture MTC 1 was isolated from a coal mine in Western Australia. Sludge and water samples were taken and used to inoculate volumes of a modified 9K medium containing yeast extract. The samples were incubated at 30°C, growth was observed after 7 days. These samples were then sub cultured in modified 9K medium without yeast extract.

Modifications and variations such as would be apparent to a skilled addressee are deemed within the scope of the present invention.

CLAIMS

1. A process for recovering metals from particulate refractory precious or base metal containing sulphide materials characterised in that it comprises contacting the sulphide material with an aqueous solution containing a thermotolerant bacteria culture (as hereinbefore defined) capable of promoting oxidation of the sulphide material at a ~~temperature in the range from 25 to 55°C,~~ separating the oxidised residue from the aqueous liquid and treating the oxidised residue and/or the aqueous liquid to recover metal therefrom.

2. A process according to Claim 1, characterised in that the aqueous solution is acidic.

3. A process according to Claim 2, characterised in that the aqueous solution has a pH in the range from 0.5 to 2.5.

4. A process according to any one of Claims 1 to 3, characterised in that the thermotolerant bacteria is acidophilic.

5. A process according to any one of the preceding claims, characterised in that the thermotolerant bacteria is aerobic.

6. A process according to Claim 5, characterised in that the aqueous liquid is aerated during the operation of the process.

7. A process according to any one of the preceding claims, characterised in that the thermotolerant bacteria is capable of autotrophic growth.

8. A process according to any one of the preceding Claims, characterised in that no Co_2 is supplied to the

thermotolerant bacteria during the operation of the process other than that available from ambient air.

9. A process according to any one of the preceding Claims, characterised in that the aqueous liquid contains sodium chloride in an amount up to 20 grams per litre.

INTERNATIONAL SEARCH REPORT

I. CLASSIFICATION OF SUBJECT MATTER (if several classification symbols apply, indicate all)[6]

According to International Patent classification (IPC) or to both National Classification and IPC
Int. Cl.[5] C22B 3/18, C12S 13/00 // C22B 11/00, 23/00 (C12S 13/00, C12R 1:01)

II. FIELDS SEARCHED

Minimum Documentation Searched[7]

Classification System	Classification Symbols
Int. Cl.[5]	C22B 3/18
Int. Cl.[4]	C11B 3/00, 11/04, 23/04

Documentation Searched other than Minimum Documentation to the Extent that such Documents are Included in the Fields Searched[8]

AU : IPC as above

III. DOCUMENTS CONSIDERED TO BE RELEVANT[9]

Category*	Citation of Document, [11] with indication, where appropriate of the relevant passages [12]	Relevant to Claim No [13]
P,X	Chemical Abstracts, Volume 115, No. 24, issued 1991, 16 December (Columbus, Ohio, U.S.A.), P.A. Spencer, J.R. Budden, and M.K. Rhodes, "Bacterial Oxidation - an econimic alterantive for the teatment of refactory gold concentrates", see page 261, column 1, abstract no. 260300n Australas. Inst. Min. Metall. Publ. Ser. 1991, (2, World Gold '91), 59-64 (Eng).	(1-9)
X	US,A,4729788 (HUTCHINS et al.) 8 March 1988 (08.03.88) See the abstract; the claims; column 3, lines 6-23.	(1-9)
X	US,A,4822413 (POOLEY et al.) 18 April 1989 (18.04.89) See column 8, lines 31-33.	(1-9)

(continued)

* Special categories of cited documents : [10]

"A" Document defining the general state of the art which is not considered to be of particular relevance

"E" earlier document but published on or after the international filing date

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"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art

"&" document member of the same patent family

IV. CERTIFICATION

Date of the Actual Completion of the International Search	Date of Mailing of this International Search Report
22 June 1992 (22.06.92)	30 June 1992 (30.06.92)
International Searching Authority	**Signature of Authorized Officer**
AUSTRALIAN PATENT OFFICE	V. THOM V.Thom

FURTHER INFORMATION CONTINUED FROM THE SECOND SHEET

X	US,A,4752332 (WU et al.) 21 June 1988 (21.06.88). See column 7, lines 7-24.	(1-9)
X	US,A,4740243 (KREBS-YUILL et al.) 26 April 1988 (26.04.88). See column 7, lines 44-61.	(1-9)
X	CA,A,1122414 (INTEROX CHEMICALS) 27 April 1982 (27.04.82). See claims 1 and 7.	(1-9)
P,X	AU,A,52258/90 (GENERAL MINING METALS AND MINERALS) 3 October 1991 (03.10.91). See claim 3.	(1-9)
P,X	US,A,5030426 (BOWERS-IRONS et al.) 9 July 1991 (09.07.91). See column 2, lines 21-34; column 4, lines 4-7.	(1-9)

V. ☐ OBSERVATIONS WHERE CERTAIN CLAIMS WERE FOUND UNSEARCHABLE [1]

This international search report has not been established in respect of certain claims under Article 17(2)(a) for the following reasons:

1. ☐ Claim numbers ...., because they relate to subject matter not required to be searched by this Authority, namely:

2. ☐ Claim numbers ...., because they relate to parts of the international application that do not comply with the prescribed requirements to such an extent that no meaningful international search can be carried out, specifically:

3. ☐ Claim numbers ...., because they are dependent claims and are not drafted in accordance with the second and third sentences of PCT Rule 6.4a

VI. ☐ OBSERVATIONS WHERE UNITY OF INVENTION IS LACKING [2]

This International Searching Authority found multiple inventions in this international application as follows:

1. ☐ As all required additional search fees were timely paid by the applicant, this international search report covers all searchable claims of the international application.
2. ☐ As only some of the required additional search fees were timely paid by the applicant, this international search report covers only those claims of the international application for which fees were paid, specifically claims:

3. ☐ No required additional search fees were timely paid by the applicant. Consequently, this international search report is restricted to the invention first mentioned in the claims; it is covered by claim numbers:

4. ☐ As all searchable claims could be searched without effort justifying an additional fee, the International Searching Authority did not invite payment of any additional fee.

Remark on Protest

☐ The additional search fees were accompanied by applicant's protest.

☐ No protest accompanied the payment of additional search fees.

ANNEX TO THE INTERNATIONAL SEARCH REPORT ON INTERNATIONAL APPLICATION NO. PCT/AU 92/00117

This Annex lists the known "A" publication level patent family members relating to the patent documents cited in the above-mentioned international search report. The Australian Patent Office is in no way liable for these particulars which are merely given for the purpose of information.

Patent Document Cited in Search Report	Patent Family Member		
US 4729788	AU 10328/88	ZA 8800071	
US 4822413	AU 69934/87 ZW 38/87	CA 1292623	ZA 8701399
US 4752332) US 4740243)	AU 62936/86		
US 5030426	AU 60378/90	US 5030425	WO 91/00369

END OF ANNEX